Biochar from Pineapple Peel as an Adsorbent for Removal of Metanil Yellow Dye in an Aqueous Solution

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DECLARATION

I declare that this thesis entitled "Biochar from Pineapple Peel as an Adsorbent for Removal Metanil Yellow Dye in an Aqueous Solution" is the result of my own research except as cited in the references.

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Bioarang daripada Kulit Nenas sebagai Penjerap untuk Penyingkiran Pewarna Kuning Metanil dalam Larutan Akueus

ABSTRAK

Pada zaman moden, industri seperti industri tekstil menggunakan pewarna secara berlebihan, yang membawa kepada pembebasan sejumlah besar air sisa yang mengandungi pewarna. Ini menimbulkan ancaman besar kepada alam sekitar dan kesihatan manusia. Walau bagaimanapun, penggunaan sisa pertanian boleh menawarkan penyelesaian yang berpatutan dan mesra alam untuk mengeluarkan pewarna dari air sisa. Kajian ini bertujuan untuk menyiasat potensi penggunaan bioarang daripada kulit nenas sebagai penjerap untuk mengeluarkan pewarna Kuning Metanil daripada larutan akueus. Kajian ini melibatkan penyediaan bioarang daripada kulit nenas menggunakan relau. Beberapa faktor yang mempengaruhi penjerapan telah diterokai, termasuk suhu karbonisasi, dos penjerap, kepekatan pewarna awal, dan masa hubungan. Keadaan optimum untuk penjerapan didapati suhu karbonisasi 700˚C, dos penjerap 1.5 g, dan kepekatan pewarna awal 50 mg/L. Kajian ini juga menekankan kepentingan masa hubungan, dengan 8 jam dikenal pasti sebagai tempoh optimum untuk mencapai kecekapan penjerapan maksimum. Model isotherm penjerapan Langmuir lebih sesuai, dengan kapasiti penjerapan maksimum 1.475 mg/g dan R^2 = 0.9985, mencadangkan proses penjerapan lapisan tunggal. Keputusan ini menunjukkan keberkesanan bioarang kulit nenas sebagai penjerap untuk mengeluarkan pewarna Kuning Metanil daripada larutan akueus.

Kata kunci: Kulit Nenas Bioarang, pewarna kuning metanil, penjerapan, Model Langmuir

Biochar from Pineapple Peel as an Adsorbent for Removal of Metanil Yellow Dye in an Aqueous Solution

ABSTRACT

In modern times, industries like the textile industry are using dyes excessively, leading to the release of large volumes of dye-containing wastewater. This poses a significant threat to the environment and human health. However, the use of agricultural waste can offer an affordable and environmentally friendly solution for removing dyes from wastewater. This study aimed to investigate the potential use of biochar from pineapple peel as an adsorbent for removing the Metanil Yellow dye from an aqueous solution. This study involved the preparation of biochar from pineapple peel using a furnace. Several factors affecting adsorption have been explored, including carbonization temperature, adsorbent dose, initial dye concentration, and contact time. The optimal conditions for adsorption were found to be a carbonization temperature of 700°C, an adsorbent dose of 1.5 g, and an initial dye concentration of 50 mg/L. The study also highlighted the significance of contact time, with 8 hours being identified as the optimal duration for achieving maximum adsorption efficiency. The Langmuir adsorption isotherm model was more suitable, with a maximum adsorption capacity of 1.475 mg/g and R^2 = 0.9985, suggesting a monolayer adsorption process. These results demonstrate the effectiveness of pineapple peel biochar as an adsorbent for removing Metanil Yellow dye from aqueous solutions.

Keywords: Biochar Pineapple Peel, Metanil Yellow dye, Adsorption, Langmuir Model

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LIST OF ABBREVIATIONS

FTIR	Fourier Transform Infrared Spectroscopy
BET	Brunauer-Emmett-Teller
MY	Metanil Yellow
GWP	Global Warming Potential

LIST OF SYMBOLS

ºC	Degree Celsius
mL	Millilitre
mg/L	Milligram per litre
nm	Nanometre
g	Gram
h	Hour
K_f	Freundlich Isotherm constant (mg/g)
C_e	Equilibrium concentration of dye in solution (mg/L)
Q_e	Amount of dye adsorbed per weight of the adsorbent at equilibrium (mg/g)
n	Adsorption intensity
q_{max}	Maximum adsorption capacity (mg/g)

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Dye is the primary material for giving the desired color to the fabric. In this regard, the coloring industry is the most important worldwide and is the main requirement in the product manufacturing process. Most of the manufacturing industries are involved with releasing waste such as producing the most frequent dyes. Moreover, the textile, clothing, and food industries use dyes to give color to the products produced. In addition, other industries that produce products such as cosmetics, rubber, turmeric powder, and paper printing also use dyes as additives. According to a study by Adane (2021), the printing and dyeing unit industry also uses the most colors in which industrial effluents released are rich in reactive dyes and chemicals.

Biochar is commonly used in the treatment of wastewater where many studies have shown that it can be produced from low-value organic matter and is often considered waste but can be converted into a more valuable product (Srivatsav et al., 2020). For example, it is obtained from shells, coconut liners, and agricultural crop residues. Several common methods are used to treat contaminated water from dye effluents flowing directly into water bodies. One of them is the adsorption process (Al-Tohamy et al., 2022). Adsorption involves the process of pulling force between the solute of adsorbate and the solid surface of the adsorbent (Yousef, 2020). In addition, there is another approach to removing the Metanil Yellow dye which is the adsorption of this dye by using biochar from pineapple peel. There are several studies for the removal of dyes using biochar from pineapple, whereby studies have been done for the removal of patent blue dyes (Kapoor et al., 2022) and the removal of methylene blue (Mahmud et al., 2021).

1.2 Problem statement

Nowadays, most of the industry uses dyeing methods and this method often uses dyes in excessive quantities and ends up being released into wastewater in large volumes. Dyes released in wastewater will pose a threat to the environment (Tripathi et al., 2023). Therefore, it affects human health through skin irritation, allergic reactions, respiratory problems, and even cancer. It also can affect other life, such as the fauna flora, as the presence of dyes in the wastewater will prevent the penetration of sunlight, thus disrupting the action of photochemistry, which creates problems with the ecological balance. The use of pineapple in the food industry is extensive, especially in the canning industry (Mardawati et al., 2023). Therefore, unused pineapple residues can cause environmental pollution problems when waste management is not properly implemented. This is because their disposal requires high costs for transportation (Aili Hamzah et al., 2021).

1.3 Objectives

The aims of this research are as follows:

1. To investigate the potential of pineapple peel-derived biochar as an adsorbent for removing Metanil Yellow dye.
2. To characterize the chemical and physical properties of pineapple peel-derived biochar through Fourier Transforms Infrared Spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET) techniques.
3. To determine the optimal adsorption parameters and the adsorption isotherm model for removal of Metanil Yellow dye using biochar derived from pineapple peel as the adsorbent.

1.4 Scope of study

This study was used pineapple peel that undergoes carbonization to become biochar as an adsorbent. Four types of adsorption parameters were investigated to identify the potential of biochar as an adsorbent for the removal of Metanil Yellow dye, which is the effect of carbonization temperature, adsorbent dosage, effect of initial dye concentration and the contact time. The characterization the chemical and physical properties of biochar-based pineapple peel was conducted using Fourier Transforms Infrared Spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET). Additionally, the adsorption isotherm models, such as Langmuir and Freundlich models, were used to analyze the adsorption behaviour of biochar.

1.5 Significant of study

The significance of this study lies in this use of biochar-based pineapple peel as an environmentally friendly adsorbent for the removal of Metanil Yellow. This is attributed to the biochar undergoes only the carbonization process and does not undergo chemical treatment. In addition, the use of biochar tends to be a carbon-rich adsorbent with a substantial surface area and porous structure, possessing the capacity to attract more dye molecules onto its surface (Patra et al., 2021). This biochar could potentially be used for the removal of dyes from industries that incorporate dyes into their products.

CHAPTER 2

LITERATURE REVIEW

2.1 Pineapple

The specific name of the pineapple fruit is *Ananas comosus* and is from the *Bromeliaceae* family (Kumar, 2023). Pineapple fruit is one of the species recognized in the genus *Ananas*. (Mohd Ali et al., 2020). Initially, pineapple originated in South Brazil and has gradually spread to other tropical regions of the world (Carvalho, 2020). In the early 1970s, Malaysia was one of the top fifth pineapple producers within Asean countries for canning purposes (Othman et al., 2011). There are several varieties of pineapple growing in Malaysia such as Josapine, Morris, MD2, Gandul, Yankee, Maspine, and Sarawak (Ismail et al., 2021). The chemical that can be found in pineapple is an enzyme called bromelain. The bromelain in pineapple has a high affinity for certain compounds, such as heavy metals and organic pollutants, and can effectively bind to them through adsorption. Pineapple also has a good source of vitamins and minerals, and it has several health benefits. Meanwhile, pineapple peel can also be used as an adsorption agent in the industry for wastewater treatment by recycling to become biochar with high carbon stability (Shakya et al., 2019). The pineapple peel can be transformed into biochar, activated carbon, and charcoal. Biochar is a type of charcoal that is useful in agriculture, as it can be added to the soil to improve soil fertility and crop yield (Kapoor et al., 2022). Activated carbon is widely used as an adsorption agent in water treatment, air purification, and food and beverage processing industries (Alves et al., 2021). Charcoal is also a useful material, especially in the production of fuel (Idowu et al., 2023). Given the potential benefits of using pineapple waste, this study aims to modify pineapple peel waste into biochar, which can be utilized in various industries.

2.2 Agriculture waste peel as an adsorbent

Agriculture is one of the most important sectors for any country, it is a source of food and raw materials for various industries. However, agriculture also contributes to the amount of agricultural waste which if waste management is not managed properly, is feared to hurt the environment. All parts of waste that are not used for human or animal food are known as agricultural waste. For example, in the process of fruit food (canning), peel and seeds from the fruit are not required in the process, including plant residues such as corn steak and cane bagasse. Usually, a few countries have used the technique to reduce all agricultural waste by open burning into lands (Tiammee et al., 2020). This action will tend to cause air pollutants such as black carbon (BC) production which can provide global warming potential (GWP) (Aakko et al., 2023). Agricultural waste such as peel also can be employed as a low-cost and effective tool in water treatment to remove the dye. A variety of agricultural waste has been applied as an adsorbent for dye removal (Table 2.1).

Table 2.1: A variety of agricultural waste has been applied as an adsorbent for dye removal.

Adsorbent	Adsorbate	Reference
Potato peel biochar	Anionic dye Cibacron Blue P3R	(Bouhadjra et al., 2021)
Litchi peel biochar	Congo red and Malachite green	(Wu et al., 2020)
Orange peel	Methylene Blue	(Giraldo et al., 2021)
Citrus Limonum (lemon) peel	Blue 49 dye	(Taifi et al., 2022)
Banana peel	Methyl orange	(Teoh et al., 2021)

2.2.1 Biochar

Biochar is a type of charcoal rich in carbon, it is produced from biomass materials such as wood chips, agricultural waste, or other organic materials through a process called pyrolysis (Amalina et al., 2022). Pyrolysis is defined as the method of thermal decomposition of biomass in the absence of oxygen to form highly carbonated biochar with porous structures (Gabhane et al., 2020). After going through the pyrolysis process, the resulting biochar becomes very porous and has a large surface area. This situation makes biochar a successful medium for removing dietary supplements from wastewater. Thus, by having high porosity properties and high adsorption, biochar can build up the expulsion of toxins in wastewater (Yaashikaa et al., 2020). Based on Rawat, (2019) study states that biochar has great potential as a sustainable and environmentally friendly solution to improve soil health and reduce waste in the biotechnology industry through adsorption.

2.3 Adsorption

Adsorption is a process that requires the use of solids to separate a substance from a solution, gas, or liquid that has usually been widely used. The separation of substances from phases through accumulation on other surfaces such as gas-solid or solid liquids, is known as the phenomenon of adsorption (Abin et al., 2022). According to a study from Aljamali (2021), this phenomenon of adsorption occurs due to the presence of residual unsaturated force fields and the lack of sufficient communication on particles on the surface. The classification of adsorption is determined based on the nature of the forces involved, it is referred to as physical adsorption or chemical adsorption. Physical adsorption occurs through weak Van der Waals forces and involves the formation of monolayers on absorbent surfaces. In contrast, chemical adsorption involves stronger chemical bonds and leads to the formation of more stable adsorption complexes.

2.3.1 Factors affecting adsorption

I. Temperature

The adsorption process is a crucial aspect of several scientific and industrial processes. It involves the uptake of one substance by another, which can occur in various scenarios. Factors such as temperature have been found to play a role in the adsorption process. According to a study by Yang et al., (2022) states that the impact of temperature on adsorption response. The study found that temperature has a relatively minor effect on the adsorption process. However, the excessive temperature increase will cause adverse effects on the adsorption process (Pernyeszi et al., 2019). The researchers found that as the temperature increases, the sorption between the adsorbate and the molecule also increases. This increase in sorption is accompanied by a positive enthalpy change (ΔH), which is considered endothermic.

II. Contact Time

The process of adsorption is one of the most important separation and purification techniques used in various industries. It involves the transfer of a substance from a fluid phase to a solid phase. The adsorption process occurs when the adsorbate molecules interact with the surface of the adsorbent. The duration for which the adsorbate and adsorbent are in contact with each other is known as contact time. It is an essential factor in the adsorption process as it determines the extent of adsorption that occurs (Al-Harby et al., 2021). During the contact time, the adsorbate molecules approach the surface of the adsorbent and stick to it. The duration of the contact time allows the adsorbate molecules to interact with the surface of the adsorbent and comply with it. Based on research from Wahyuhadi et al., (2023) states that increased contact time affects the increase in absorption capacity. This is because as the contact time increases, more adsorbate molecules can approach the adsorption surface, then induce the adsorbate to spread widely and lead to increased adsorption.

III. Particle size of adsorbent

The particle size of the adsorbent material plays an important role in determining the efficiency and effectiveness of the adsorption process. This is because the particle size of the adsorbent greatly affects the adsorption capacity, rate, and balance achieved during the process (Sudrajat et al., 2021). Based on the study from Yuan et al., (2021), the change in adsorption capacity occurs due to changes in the surface area of a particular biochar. The surface area of the adsorbent is directly proportional to the size of its particles. Finer particles have a greater surface area compared to abrasive particles. As the surface area increases, then more active sites are available for adsorption.

IV. Concentration of Adsorbate

The concentration of adsorbate plays an important role in influencing the adsorption process. Adsorption refers to the state of molecules or particles attached from the adsorbate phase to the adsorbent surface area. This phenomenon occurs due to the force between molecules, such as Van der Waals or chemical bonds, between adsorbent and adsorbate. A study by Blachnio et al, (2020) states that the interaction properties of adsorbate molecules are necessary and are an important factor in obtaining a relative adsorption rate.

2.4 Adsorption Isotherm

Adsorption isotherm is a fundamental concept in the field of adsorption, which describes the reaction between the amount of adsorbate and its concentration in the adsorbent at a given temperature (Raji et al., 2023). It provides valuable knowledge about the adsorption process and helps in understanding the interaction between the adsorbate and adsorbent. In addition, it is typically represented graphically, with the amount of adsorbate adsorbed on the y-axis and the concentration or pressure of the adsorbate on the x-axis. The adsorption isotherms pertain to the adsorption of a single layer of adsorbate, where the adsorption enthalpies remain constant, and densely packed molecules necessitate the consideration of interaction energies throughout the adsorption process (Murphy et al., 2023).

Different types of adsorption isotherms have been identified based on the behavior exhibited by adsorbents under specific conditions (Kalam, 2021). One commonly used adsorption isotherm model is the Langmuir isotherm, which assumes monolayer adsorption on a homogeneous adsorbent surface (Lopez et al., 2019). According to the Langmuir model, adsorption occurs through the formation of a reversible equilibrium between the adsorbate molecules in the bulk phase and the adsorbed molecules on the surface. Another commonly used model is the Freundlich isotherm, which is an empirical equation that does not assume a specific adsorption mechanism (Na, 2020).

2.5 Previous of metanil yellow dye

Metanil Yellow (MY) is a synthetic dye belonging to the azo dye class. It is also known by other names such as Yellow Acid 36 or C.I 13065. The Metanil Yellow molecular formula C18H14N3NaO3S, Metanil Yellow is widely used in various industries, especially in the textile, plastic, and food industries, as a dye for fabrics, plastic products, and food goods (Kourani et al., 2020). However, it has been banned in a few countries due to concerns about its safety and potential health risks (Khan et al., 2020). Figure 2.5 shows the structure of Metanil Yellow.

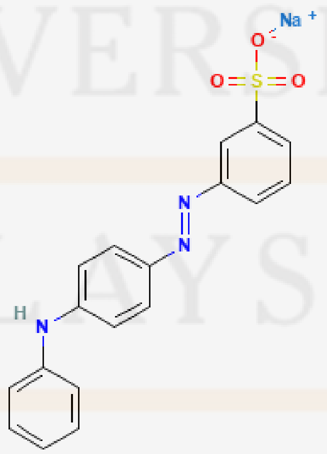

Figure 2.1: Structure of Metanil Yellow

(Source: National Center for Biotechnology Information, 2023)

CHAPTER 3

MATERIALS AND METHODS

3.1 Material

The material that was used in this study was 60g of pineapple peel. The pineapple was obtained from Pantai Timur market, Jeli. The preparation process requires the utilization of pineapple peel waste to create biochar, while the Metanil Yellow dye (R&M Chemicals) was utilized as an adsorbate. Distilled water was also employed for the preparation of the solutions.

3.1.1 Equipment and apparatus

The apparatus that was used for this study includes various vessels such as a beaker (50 mL and 100mL), a volumetric flask (50mL, 100 mL and 500 mL), a conical flask (250 mL), as well as reagent bottles (100 mL and 1000 mL). Other items such as filter funnel, filter paper, cuvette, stainless steel sieve (125 µm), glass rod, and spatula was required. Furthermore, certain instruments were utilized: a weighing balance, a sieving machine, a furnace, a UV-Vis Spectrophotometer, and a blender.

3.2 Methods

The method used in this study was the preparation of biochar from pineapple peel. The following method was the preparation of adsorbate by using metanil yellow powder and preparation a calibration curve using metanil yellow solution.

3.2.1 Preparation of adsorbent (Biochar)

The fresh fruit pineapple was bought from Pantai Timur market, Jeli. The pineapple was rinsed with water to remove dirt and clean the surface of the fruit. Then, the peel was chopped into small pieces and dried in the oven at 60˚C to remove moisture (Shakya et al., 2019). The peel of the pineapple was blended and sieved into 125 µm. Then, 40 grams of peel was placed into the crucible and was carbonized using furnace at 400˚C and 700˚C for 2 hours (Gan, 2021). This crucible was placed in a furnace for carbonization in atmospheric air. The biochar derived from the pineapple peel was placed in airtight plastic containers and stored at room temperature for future use.

3.2.2 Preparation of adsorbate (Metanil Yellow Stock Dye Solution)

The chemical used in this study was Metanil Yellow ($C_{18}H_{14}N_3NaO_3S$), also called Acid Yellow 36. The stock solution was prepared by dissolving 0.5 g of dye powder in distilled water using a 500 mL volumetric flask (Rao et al., 2020). The stock solution of Metanil Yellow was prepared with a concentration of 1000 mg/L (Sivashankar et al., 2022).

3.2.3 Preparation of a calibration curve

The process involves diluting a stock solution of Metanil Yellow dye with distilled water in a 100 mL volumetric flask at various concentrations of 0.5, 2.0, 4.0, 6.0, 10.0, and 20.0 mg/L (Oetjik et al., 2021). A blank solution with no dye (0 mg/L) was prepared using distilled water. The blank and stock solutions were then placed in a UV-Vis spectrophotometer set at a wavelength of 434 nm (Rizvi et al., 2022)). The absorbance reading obtained from the spectrophotometer was used to construct the calibration curve for Metanil Yellow.

3.3 Adsorption parameter

In this section, the investigation was involved the factors of the adsorption parameter, such as influence of carbonization temperature, adsorbent dosage, initial dye concentration, and contact time.

3.3.1 The effect of carbonization temperature

The effects of carbonization temperature were studied at two temperatures specifically at 400˚C and 700˚C using a furnace (Song et al., 2019). The pineapple peel at 125 µm was put into several crucibles to form biochar.

3.3.2 The effect of adsorbent dosage

The effect of adsorbent dosage was analyzed with different amounts of dosage (0.5, 0.7, 0.9, 1.2, 1.5, 2.0 and 2.5 g) over 24 hours at ambient temperature (Elkhaleefa et al., 2021). This study used a biochar at the temperature of 700˚C. This mixture of dye and adsorbent was stirred using a glass rod for a few minutes and rested for 24 hours.

3.3.3 The effect of Initial dye concentration

The effect of the initial concentration of Metanil Yellow was examined by using concentrations at 20, 50, 100, 150, 200, and 250 mg/L over 24 hours at room temperature (Oetjik et al., 2021). The study used an adsorbent dosage of 1.5 g. The dye and adsorbent mixture were stirred with a glass rod for a few minutes to ensure proper mixing, followed by a 24-hour resting period. The initial dye effect was analyzed using the optimum carbonization temperature (700˚C) and adsorbent dosage (1.5 g) data that were obtained from previous experiment.

3.3.4 The effect of contact time (24hours)

The effect of contact time was assessed at 1 h, 3 h, 5 h, 8 h, 10 h, and 12 h (Kurniawati, 2021). This experiment was carried out by using optimum carbonization (700˚C), adsorbent dosage (1.5 g) and initial dye concentration (50 mg/L) obtained in the previous experiments.

3.4 Characterization Technique of Adsorbent

This study used two samples such as biochar pineapple peel and biochar pineapple peels that were absorbed by Metanil Yellow dye and were analyzed using infrared transform Fourier, FTIR equipment, and Brunauer-Emmett-Teller (BET). The FTIR was used to identify functional groups in the biochar sample, while BET equipment was used to investigate the sample's surface area.

3.5 Data analysis

Two specific equations were utilized for data analysis by following the Langmuir isotherm equation and the Freundlich isotherm equation. This study was analyzed using the Langmuir isotherm, and Freundlich isotherm equations.

3.5.1 Langmuir adsorption model

$$q_e = \frac{q_{max}\, K_L\, C_e}{1+K_L C_e}$$ Equation 3.1

i. Linearised Langmuir Isotherm following equation,

$$\frac{C_e}{q_e} = \frac{1}{q_{max} K_L} + \frac{1}{q_{max}}\, C_e$$ Equation 3.2

Where:

q_e = Amount of metanil yellow adsorbed (mg/g^{-1})

q_{max} = maximum adsorption capacity (mg/g^{-1})

K_L = Langmuir adsorption constant (L/mg^{-1})

C_e = Equilibrium metanil yellow concentration (mgL^{-1})

3.5.2 Freundlich adsorption model

$$Q_e = K_f C_e^{\frac{1}{n}}$$ Equation 3.3

Where:

n = Adsorption intensity

K_f = Freundlich isotherm constant (mg/g)

Ce = Equilibrium concentration of dye in solution (mg/L)

Qe = Amount of dye adsorbed per weight of the adsorbent at equilibrium (mg/g)

ii. Linearised Freundlich Isotherm

$$logQ_e = \frac{1}{n}\ logC_e + logK_f$$ Equation 3.4

Where:

K_f = Freundlich isotherm constant (mg/g)

1/n = Empirical parameter that relate to the adsorption intensity

CHAPTER 4

RESULT AND DISCUSSION

4.1 Characterization of biochar pineapple peel.

This study focuses on the conversion of pineapple peel into biochar, which can be used as an adsorbent to remove Metanil Yellow dye. The adsorbents were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and Brunauer-Emmett-Teller (BET) transforms.

4.1.1 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The effects of Metanil Yellow dye adsorption on the FTIR spectrum of biochar pineapple peel were examined. The FTIR spectra were measured in the range of 500 cm^{-1} to 4000 cm^{-1}. Figure 4.1 displays the results of this analysis, where Sample 1 represents the spectrum of biochar pineapple peel before the Metanil Yellow dye adsorption, while Sample 2 represents the spectrum after the adsorption. During the FTIR analysis, no functional group was detected, but this does not necessarily mean that there was no peak. In FTIR analysis, sometimes the peak of a functional group cannot be detected due to various reasons. This may happen due to the low concentration of the functional group in the sample or if the path length of the sample is too short (Li et al., (2016). Additionally, the peak may be too weak to detect if the functional group is present in a complex mixture of compounds (Nori et al., (2019).

According to Fu et al. (2016), the functional groups identified in the biochar from pineapple peel included hydroxyl groups (O-H), aliphatic C-H groups, carbonyl (C=O) groups, aromatic compounds, and alcohols/phenolics. These functional groups contribute to the sorption properties of the biochar, making it effective for adsorbing dyes. Shakya (2019) reported that the raw pineapple peel showed peak at approximately 3295 cm^{-1} due to the hydrogen-bound O-H stretch that corresponding to the cellulose and hemicellulose substances. However in this study, the pineapple peel has undergo the carbonization process to produce biochar, therefore the peak was disappeared in pineapple peel biochar which indicated the elimination of hemicellulose and cellulose after the carbonization of pineapple peel (Elean et al 2022). The findings suggest that the thermal degradation of pineapple peel at higher temperatures leads to changes in the chemical composition of the biochar, such as hydroxyl groups (O-H), aliphatic C-H groups, carbonyl (C=O) groups, aromatic compounds, and alcohols/phenolics. These changes can be detected through FTIR analysis.

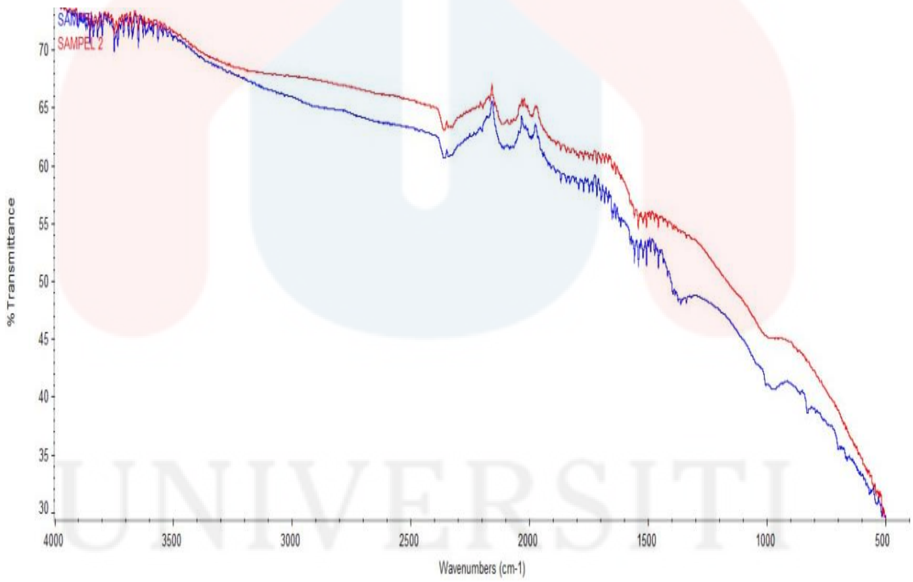

Figure 4.1 FTIR spectra for biochar pineapple peel (700 ˚C) before and after dye adsorption.

4.1.2 Brunauer-Emmett-Teller (BET) Analysis

In this study, the BET analysis was used to measure the surface area of a biochar by adsorbing a gas onto its surface and measuring the amount of gas adsorbed at different pressures. The surface area was then calculated based on the amount of gas adsorbed and the pressure at which it was adsorbed.

Table 4.1: The BET surface area for biochar before and after adsorption

Biochar Pineapple Peel	**BET surface area (m^2/g)**
Before adsorption	27.288
After adsorption	1.108

Table 4.1 displays the results of the BET analysis conducted on the biochar sample before and after the adsorption process. The results indicate that the biochar's surface area reduced significantly after removing the Metanil Yellow dye. Specifically, the surface area was decreased from 27.288 m^2/g to 1.108 m^2/g. This is because the Metanil Yellow dye molecule clogs the pores during adsorption, reducing the biochar's surface area ((Han et al., 2021). According to Eleryan et al (2022), the study also found that the surface area of Mandarin Biochar-TETA (MBT) decreased from 5.48 m^2/g to 2.01 m^2/g after the Acid Yellow 11 dye adsorption. This decrease was attributed to the blocking of the pores and the reduction of the surface functional groups of MBT by the dye molecules.

Therefore, this result showed that the biochar derived from agricultural waste, such as pineapple peel, highlights the importance of understanding the surface properties of biochar for effective adsorption applications. The decrease in BET surface area after adsorption was a crucial factor to consider in the practical application of biochar for adsorption purposes (Suhaimi et al., 2022). It indicates the need for regeneration or replacement of the biochar after it has been used for adsorption to maintain its effectiveness as an adsorbent (Iamsaard et al., 2022).

4.2 Calibration curve

In this study, a series of solutions were prepared using a stock solution of Metanil Yellow with a concentration of 1000 mg/L. The solutions were prepared with varying concentrations ranging from 0.5 mg/L to 20 mg/L. This concentration range was selected to create a linear calibration curve that can be used to determine the correlation between the concentration of the dye and the absorbance reading by the solution (Besegatto, 2020). The calibration curve for Metanil Yellow is shown in Figure 4.2. The coefficient value obtained from the calibration curve showed a good result of 0.999. The correlation coefficient (R^2) was used as a benchmark to measure the accuracy of this study. According to Sharma et al., (2019), an R^2 value that is nearly 1 indicates a strong correlation. The calibration curve was useful in determining the final concentration of the dye present in the solution after it had undergone adsorption by biochar (Chen, 2022). The adsorption process involves the extraction of the dye from the solution by biochar, which can be used to estimate the quantity of dye removed. The curve depicts a linear relationship between the dye's concentration and the light's absorbance by the solution at 434 wavelengths using a spectrophotometer.

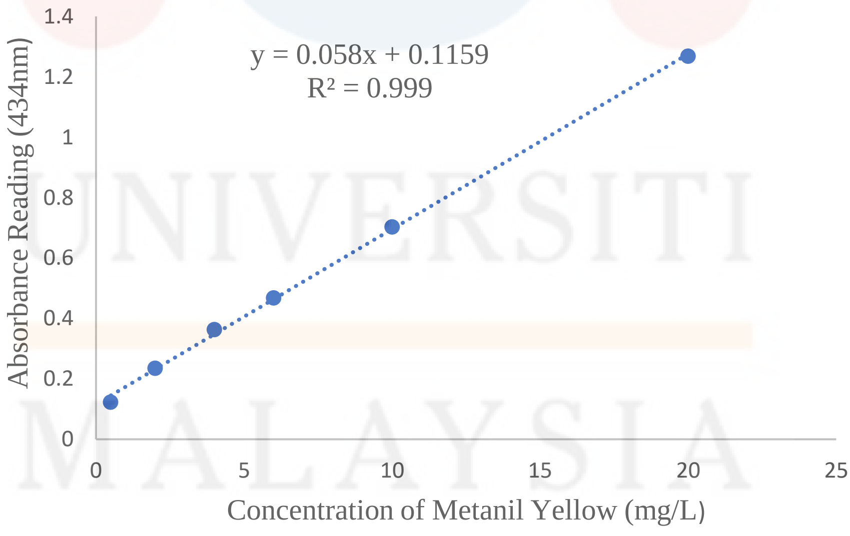

Figure 4.2: Calibration curve of Metanil Yellow dye at 434nm.

4.3 Effect of Carbonization Temperature

The data presented in Figure 4.3 shows the most effective carbonization temperature is 700˚C, with a 55.15% removal rate. This temperature results in a higher percentage of removal in comparison to 400˚C. The percentage removal at 700˚C is significantly greater, making it the optimal temperature for carbonization. On the other hand, at 400˚C, the percentage removal is only 6.54%, which is much lower than that achieved at 700˚C. As the carbonization temperature increases, it leads to a higher surface area and porosity of biochar. This is due to the loss of more volatile matter and the creation of more micro pores during the carbonization process at higher temperatures (Goswami et al., 2022). Thus, the yield of biochar will also decrease due to the loss of volatile substances and gas emissions. A higher carbonization temperature produces increased carbon content, aromatic structures, and decreased polarity, all of which contribute to an increased adsorption capacity for removing Metanil Yellow dye (Woo et al., 2021).

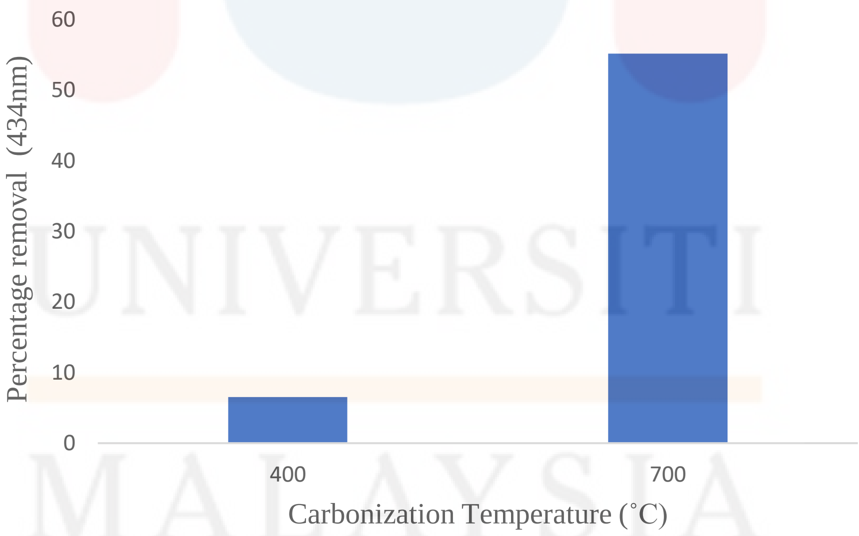

Figure 4.3: Effect of carbonization temperature on the adsorbent. Condition: adsorbent dosage 0.5 g, initial dye concentration 50 mg/L, contact time 5 h, at room temperature.

4.4 Effect of Adsorbent Dosage

Figure 4.4 shows the effect of the adsorbent dose on the removal of the Metanil Yellow dye has increased from 0.5 g to 1.5 g and after that continues to be constant to 2.5 g. The optimal value of the effect of the adsorbent dose is 1.5 g with a percentage removal of 92.40%. The relationship between the adsorbent dose and the percentage of removal increases with an increase in the dose of adsorbent (Zhong et al., 2022). This is due to the increased availability of active sites on the surface of the adsorbent, which leads to a higher number of absorbed molecules, resulting in a more significant percentage of removal (Luo et al., 2021). However, when the adsorbent becomes saturated with dye molecules, a further increase in the dose of adsorbents may not lead to additional removal, resulting in a constant percentage of removal (Nagappan et al., 2019). This phenomenon is associated with an adsorbent that reaches its maximum adsorption capacity. Thus, the initial increase and subsequent plateau in the removal of percentages with an increase in the dose of the adsorbent can be understood in the context of the adsorption process and saturation of the active site of the adsorbent.

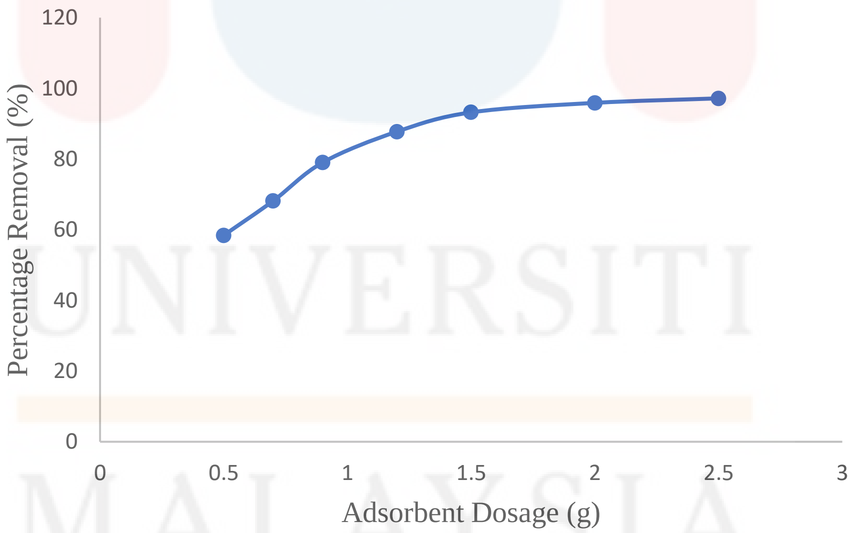

Figure 4.4: Effect of adsorbent dosage on removal of Metanil Yellow dye. Condition: Initial concentration dye 50 mg/L, contact time 5 h, at room temperature.

4.5 Effect of Initial Metanil Yellow Dye Concentration

The effect of this initial concentration of Metanil Yellow dye is one of the important factors affecting the adsorption process. For this part, the dose of biochar used was 1.5 g for each initial concentration of Metanil Yellow dye, which varies from 20 to 250 mg/L. Based on Figure 4.5, the graph shows a decrease in the percentage of removal starting from 96.56% to 75.49% as the concentration increases. This is because 1.5 g of biochar used has reached saturation levels where higher concentrations of Metanil Yellow dyes lead to more dye molecules in the solution, which can weave the available site faster. According to a study by Nizam et al., (2021) states that the active site will be limited as the initial concentration of the dye increases because it will contain a large number of molecules resulting in competition for the same number of active sites. In this study, the optimum initial dye concentration was 50 mg/L with a removal percentage of 91.58%. This is because at a concentration of 50 mg/L, the percentage of dye removal stabilizes at around 70% and remains constant even if the concentration of dye changes (Jóźwiak et al., 2020).

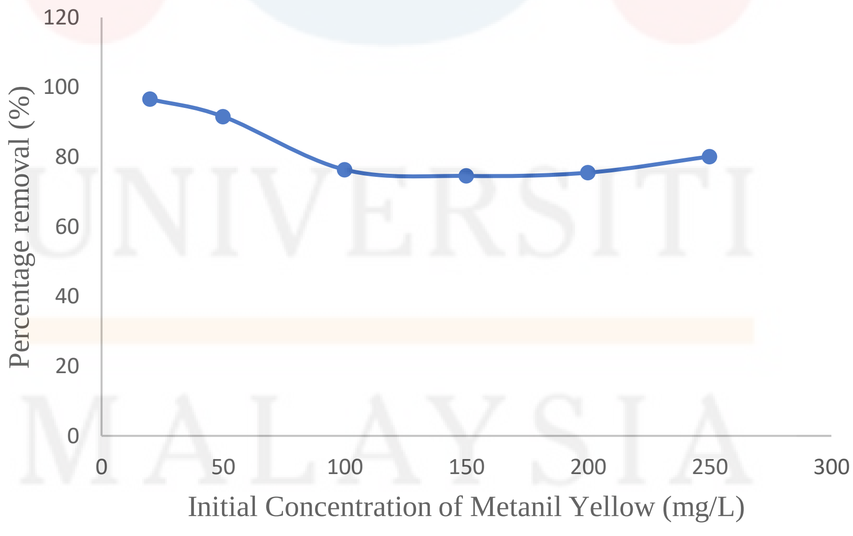

Figure 4.5: Effect of initial Metanil Yellow concentration. Condition: adsorbent dosage 1.5 g, contact time 5 h, at room temperature.

4.6 Effect of Contact Time

Adsorption studies were conducted at various contact times of 1, 3, 5, 8, 10, and 12 hours. The initial concentration of the dye used was 50 mg/L, with a 1.5 g adsorbent dose of biochar pineapple peel in a 50 mL dye solution of Metanil Yellow. The graph in Figure 4.6 illustrates how the adsorption of Metanil Yellow dyes onto biochar from pineapple peel was affected by contact time. The data showed that the adsorption of Metanil Yellow increased with contact time up to 8 hours, after which no further increase was observed. It can be explained that the initial increase in adsorption occurs because of the existence of active sites on biochar pineapple peel (Tsai et al., 2022). However, as time passes, these sites become saturated, which leads to a plateau in adsorption efficiency (Safri et al., 2022). Therefore, the optimal contact time for maximum adsorption efficiency has been determined to be 8 hours. This is because if the duration exceeds this limit, the number of active sites does not proportionally match the amount of dye, which results in no significant increase in adsorption. Based on the results, the optimum conditions of the adsorption process were achieved after 8 hours.

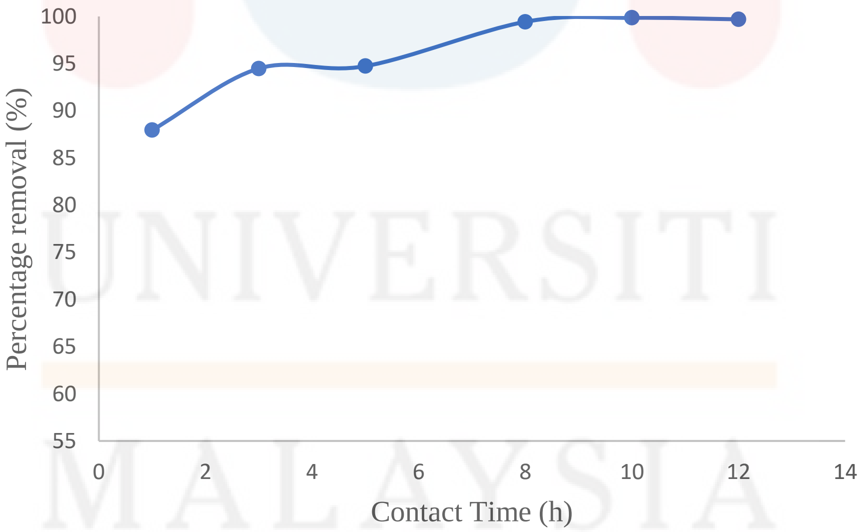

Figure 4.6: Effect of Contact Time. Condition: adsorbent dosage 1.5 g, initial dye concentration 50 mg/L, at room temperature.

4.7 Adsorption Isotherm

In this study, the Langmuir and Freundlich models were used to know the behavior of the adsorption process of the Metanil Yellow dye. The result of these isotherms for the adsorption of Metanil Yellow dye were presented in Table 4.2, the Langmuir isotherm revealed a maximum adsorption capacity (qmax) of 1.475 mg/g and a Langmuir constant (K_L) of -16.222 L/mg, with a correlation coefficient (R^2) value of Langmuir is 0.9985. On the other hand, the Freundlich isotherm showed a Freundlich constant (K_F) of 2.216 L/mg, a non-linearity factor (n) of -40.650, and a correlation coefficient (R^2) value of 0.8117.

The graph presented in Figure 4.6 shows that the Metanil Yellow dye is highly attracted to biochar, which can be evidenced by its maximum adsorption capacity of 1.475 mg/g. The Langmuir model was found to be the most suitable, as indicated by the high correlation coefficient (R^2) value of 0.9985, which suggests that the adsorption occurs uniform surface in a monolayer manner. This means that the Metanil Yellow dye molecules form a single layer on the biochar surface (Khayyun et al., 2019).

According to Figure 4.7, the Freundlich model shows that biochar has a strong adsorption capacity (Kf) of 2.216 mg/g, meaning that it has a high affinity for dye. However, the relationship between the two is non-linear. The negative value of n indicates an unprofitable adsorption process, and the lower R^2 value of 0.8117 suggests that the equilibrium data was not fit to the Freundlich model. These results are consistent with the adsorption behavior of Metanil Yellow reported in the literature. For example, a study on the removal of Metanil Yellow from aqueous solutions using micro-nano silica particles (MNSPs) found that the adsorption behavior followed the Langmuir model, with an R^2 value close to 1, indicating linear adsorption of Metanil Yellow by the adsorbent (Nagappan et al., 2019). Therefore, it can be concluded that the Langmuir model is a better fit for this study than the Freundlich model.

Table 4.2: Adsorption isotherm parameter based on Langmuir Isotherm and Freundlich Isotherm

Isotherm Model	Parameter Values
Langmuir	
q_{max} (mg/g)	1.475
K_L (L/mg)	-16.222
R^2	0.9985
Freundlich	
K_F (mg/g)	2.216
n	-40.650
R^2	0.8117

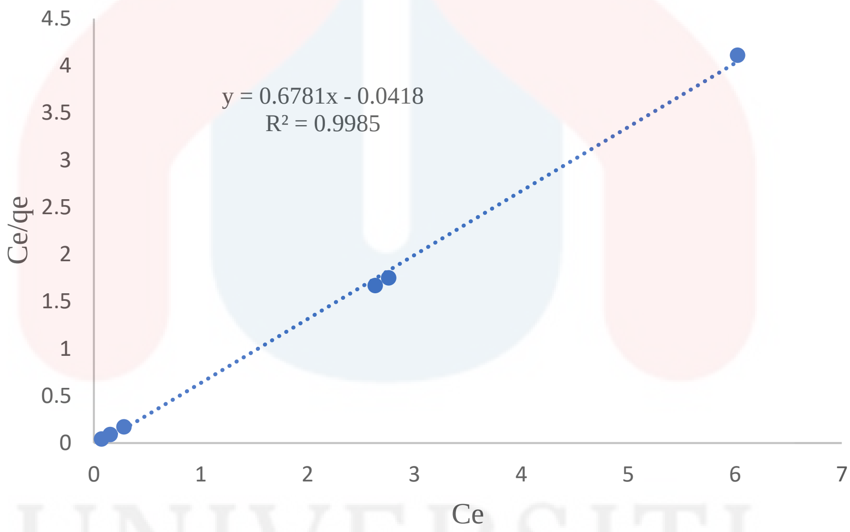

Figure 4.7: Langmuir plot for contact time of pineapple peel biochar onto Metanil Yellow dye.

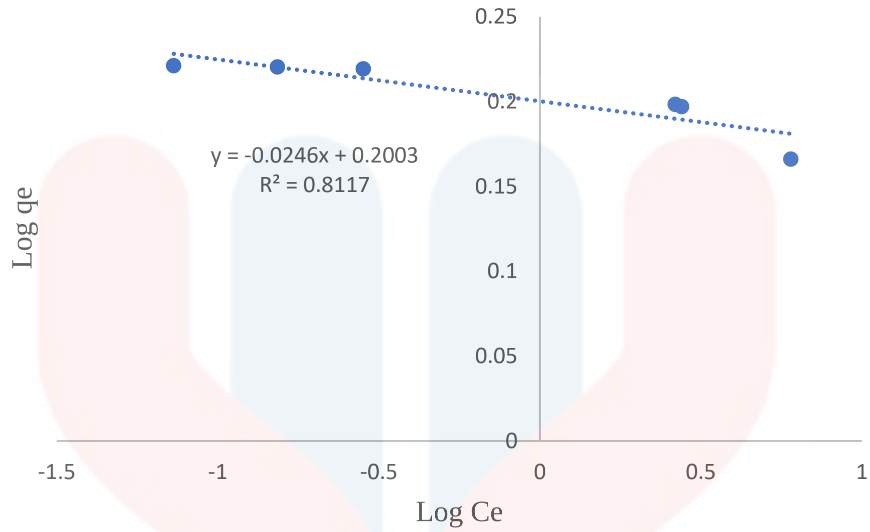

Figure 4.8: Freundlich plot for contact time of pineapple peel biochar onto Metanil Yellow dye.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This study concluded that biochar derived from pineapple peel has great potential as an effective adsorbent for the removal of Metanil Yellow dye. It has been revealed that pineapple peel biochar has a high adsorption capacity and can remove up to 55.15% of the Metanil Yellow dye.

The FTIR analysis indicates that the carbonization process has resulted in the absence of functional groups due to high-temperature conditions. However, prior research has demonstrated the presence of several functional groups such as hydroxyl, aliphatic C-H, carbonyl, aromatic compounds, and alcohols/phenolics in biochar derived from pineapple peel. These functional groups contribute to the biochar's sorption properties for dye removal. Besides, through the BET analysis was proven that biochar was effective in adsorbing Metanil Yellow dye. This is evidenced by a significant decrease in surface area after dye adsorption, which indicates that the dye molecules are blocking the pores.

This study identified the optimal parameters for removing Metanil Yellow dye through adsorption. The best results were achieved with a carbonization temperature of 700°C, an adsorbent dose of 1.5 g, and an initial dye concentration of 50mg/L. The adsorption of Metanil Yellow dye increased up to 8 hours of contact time considered as optimum parameter. The Langmuir model was found to be the most suitable for describing the adsorption isotherm model, with a high correlation coefficient (R^2) value of 0.9985. This suggests uniform surface adsorption in a monolayer manner.

5.2 Recommendation

The study has provided several recommendations for further research and practical applications of pineapple peel biochar. The first recommendation is to optimize the carbonization temperature of pineapple peel biochar to increase its adsorption capacity for Metanil Yellow dye. The study suggests that the adsorption capacity of pineapple peel biochar can be further improved by optimizing the temperature at which it is produced. By increasing the temperature, the surface area of the biochar can be increased, which in turn can enhance its adsorption capacity (Patra et al., 2021).

Practical applications of pineapple peel biochar should also be investigated, particularly in real-world scenarios such as wastewater treatment plants, to assess the scalability and effectiveness of pineapple peel biochar for dye removal. This could involve testing the effectiveness of pineapple peel biochar in real-world conditions and evaluating the feasibility and cost-effectiveness of using it for dye removal (Kapoor et al., 2022).

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APPENDIX A

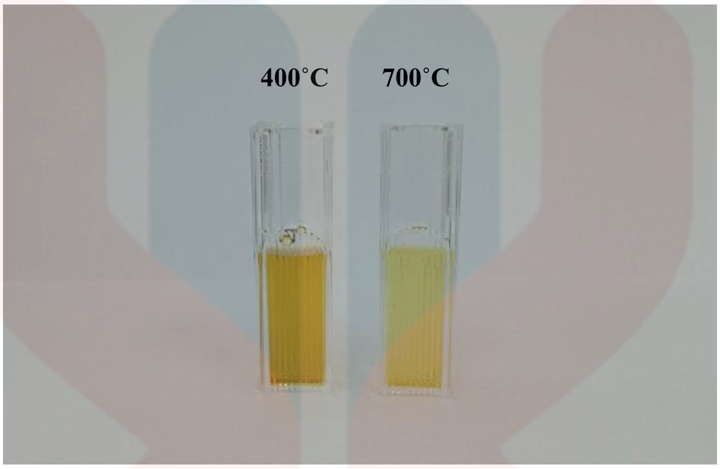

Figure A1: Effect of carbonization temperature on adsorption

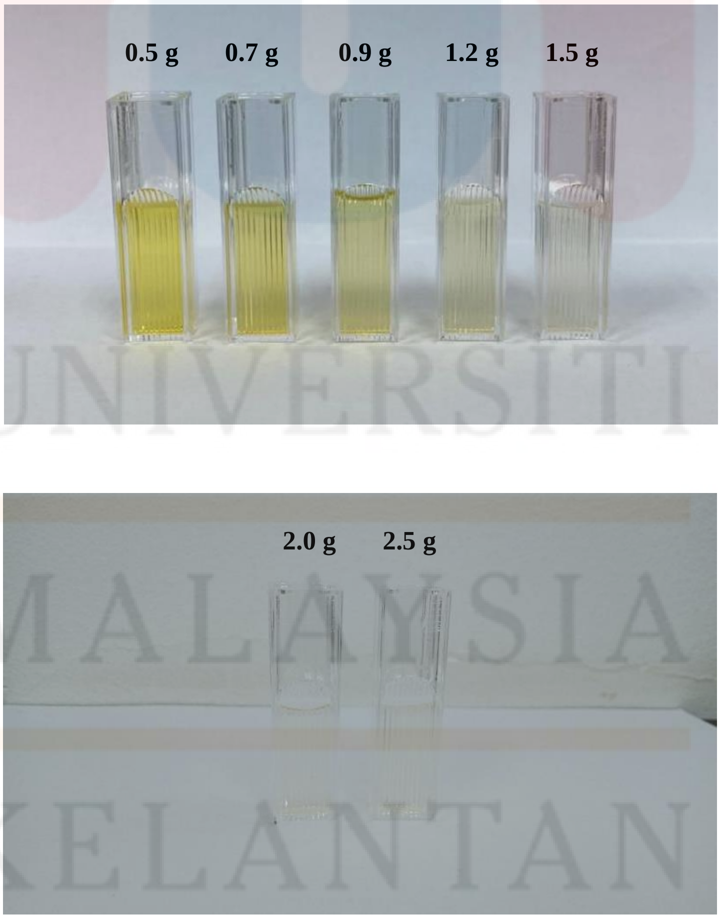

Figure A2: Effect of adsorbent dosage on adsorption

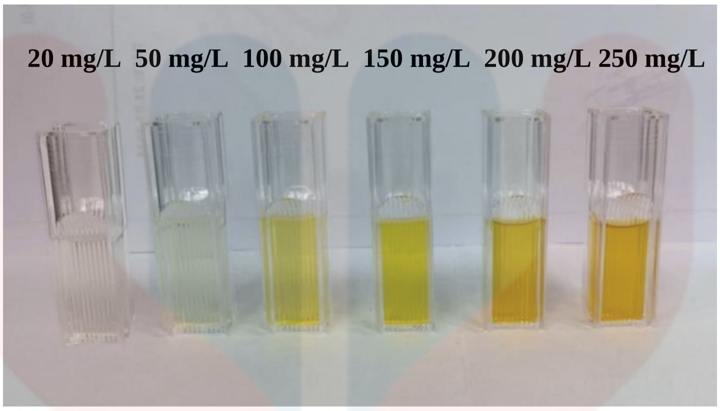

Figure A3: Effect of initial dye concentration on adsorption

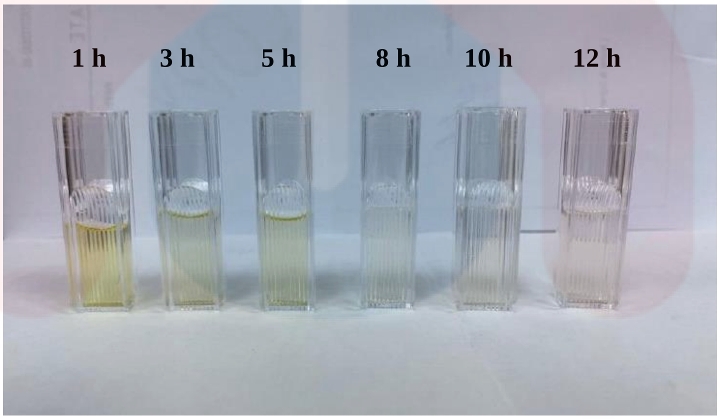

Figure A4: Effect of contact time on adsorption

APPENDIX B

Table B.1: Data for Calibration Curve of Metanil Yellow Dye

Concentration of MY (mg/L)	Absorbance Reading 1	Absorbance Reading 2	Absorbance Reading 3	Average	Standard Deviation
0.5	0.123	0.133	0.123	0.126	0.0047
2	0.235	0.242	0.251	0.242	0.0065
4	0.363	0.374	0.363	0.366	0.0052
6	0.468	0.472	0.484	0.474	0.0068
10	0.703	0.709	0.714	0.708	0.0045
20	1.268	1.274	1.283	1.275	0.0062

Table B.2: Data for Carbonization Temperature of Adsorbent

Carbonization Temperature (˚C)	Abs R1	Abs R2	Abs R3	Average	Ce (mg/L)	Percent age Remov al (%)
400	2.644	2.971	2.864	2.826	46.731	6.536
700	1.200	1.860	1.190	1.416	22.427	55.146

Table B.3: Data for Absorbent Dosage

Absorbent Dosage (g)	Abs R1	Abs R2	Abs R3	Average	Ce (mg/L)	Percentage Removal (%)
0.5	1.322	1.421	1.322	1.355	21.363	57.272
0.7	1.039	1.042	1.054	1.045	16.018	67.962
0.9	0.724	0.736	0.740	0.733	10.645	78.709
1.2	0.471	0.486	0.501	0.486	6.381	87.237
1.5	0.313	0.340	0.356	0.336	3.800	92.398
2.0	0.235	0.248	0.252	0.245	2.225	95.548
2.5	0.198	0.201	0.219	0.206	1.553	96.893

Table B.4: Data for Initial Concentration of Adsorbate

Initial Concentration (mg/L)	Abs R1	Abs R2	Abs R3	Average	Ce (mg/L)	Percentage Removal (%)
20	0.076	0.124	0.235	0.145	0.501	97.491
50	0.360	0.382	0.396	0.379	4.541	90.916
100	1.488	1.493	1.488	1.489	23.685	76.314
150	2.330	2.346	2.357	2.344	38.421	74.385
200	2.959	2.972	2.963	2.963	49.093	75.453
250	3	3	3	3	49.725	80.109

Table B.5: Data for Contact Time of Adsorption

Contact Time (hours)	Abs R1	Abs R2	Abs R3	Average	Ce (mg/L)	Percentage Removal (%)
1	0.451	0.467	0.478	0.465	6.024	87.950
3	0.261	0.288	0.288	0.276	2.760	94.479
5	0.267	0.267	0.267	0.268	2.633	94.732
8	0.125	0.133	0.139	0.132	0.283	99.433
10	0.101	0.112	0.122	0.112	0.073	99.854
12	0.096	0.106	0.119	0.107	0.153	99.693

APPENDIX C

Table C.1: Equilibrium Data for Adsorption Isotherm

Contact Time (hours)	Ce (mg/L)	qe	Ce/qe	Log Qe	Log Ce
1	6.025	1.466	4.111	0.166	0.779
3	2.760	1.575	1.753	0.197	0.441
5	2.634	1.579	1.669	0.198	0.420
8	0.283	1.657	0.171	0.219	-0.547
10	0.073	1.664	0.044	0.221	-1.136
12	0.153	1.662	0.092	0.220	-0.814